Microscale Acid-Base Titration

A titration is a process used to determine the volume of a solution needed to react with a given amount of another substance. In this experiment, you will titrate hydrochloric acid solution, HCl, with a basic sodium hydroxide solution, NaOH. The concentration of the NaOH solution is given and you will determine the unknown concentration of the HCl. Hydrogen ions from the HCl react with hydroxide ions from the NaOH in a one-to-one ratio to produce water in the overall reaction:

$$H^+(aq) + Cl^-(aq) + Na^+(aq) + OH^-(aq) \longrightarrow H_2O(l) + Na^+(aq) + Cl^-(aq)$$

When HCl solution is titrated with NaOH solution, the pH value of the acidic solution is initially low. As base is added, the change in pH is quite gradual until close to the equivalence point, when equimolar amounts of acid and base have been mixed. Near the equivalence point, the pH increases very rapidly. The change in pH then becomes more gradual again, before leveling off with the addition of excess base.

Since this experiment may be your introduction to acid-base titrations, you will determine only the *approximate* concentration of the hydrochloric acid solution. Use the formula:

$$M_{acid} = M_{base} \times \frac{V_{base}}{V_{acid}}$$

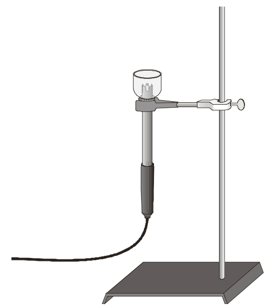
where M_{acid} is the concentration of the acid (in M or mol/L), M_{base} is the concentration of the base, V_{base} is the volume of the base (in drops), and V_{acid} is the volume of the acid. The concentration of the sodium hydroxide solution is 0.10 M. The drops of sodium hydroxide and hydrochloric acid solutions at the equivalence point will be determined from the experiment.

MATERIALS

LabPro or CBL 2 interface
TI Graphing Calculator
DataMate program
pH Sensor
wash bottle
distilled water
toothpick (for stirring)
0.10 M NaOH solution (in dropper bottle)
HCl solution (in a dropper bottle)
ring stand
utility clamp
phenolphthalein indicator
micro-beaker (top half of a storage bottle for the pH Sensor)

PROCEDURE

1. Obtain and wear goggles.
2. Prepare the pH Sensor for data collection.
   a. Plug the pH Sensor into Channel 1 of the LabPro or CBL 2 interface. Use the link cable to connect the TI Graphing Calculator to the interface. Firmly press in the cable ends.
   b. Remove the pH Sensor from the pH storage solution bottle by unscrewing the lid. Carefully slide the lid from the sensor body.
   c. Rinse the tip of the sensor with distilled water.



d. Obtain a pH Sensor storage bottle that has been cut in half. This is your microbeaker!

e. With the open end of the pH Sensor pointing upward, as shown here, slip the microbeaker and cap down onto the sensor body (small opening first), so the sensor tip extends about 1 cm into the bowl of the microbeaker. Then tighten the threads of the cap so the cap tightens snugly against the pH Sensor body.

f. Attach the utility clamp to a ring stand and to the bottle lid, with the sensor in an inverted position as shown here.

3. Obtain a dropper bottle containing the HCl solution of unknown concentration. Add 10 drops of the HCl solution into the micro-beaker. As you add the drops, hold the bottle in a vertical position to ensure that drop size is uniform. **CAUTION:** *Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin.* Add 1 drop of phenolphthalein indicator to the microbeaker, then add enough distilled water so the resulting solution completely covers the sensor tip. Stir the solution thoroughly with the toothpick.

4. Obtain a dropper bottle containing 0.10 M NaOH. Wait until Step 8 to begin adding this solution to the HCl solution in the microbeaker.

5. Turn on the calculator and start the DATAMATE program. Press CLEAR to reset the program.

6. Set up the calculator and interface for the pH Sensor.

   a. Select SETUP from the main screen.

   b. If CH 1 displays PH, proceed directly to Step 7. If it does not, continue with this step to set up your sensor manually.

   c. Press ENTER to select CH 1.

   d. Select PH from the SELECT SENSOR menu.

7. Set up the data-collection mode.

   a. To select MODE, press ▲ once and press ENTER.

   b. Select EVENTS WITH ENTRY from the SELECT MODE menu.

   c. Select OK to return to the main screen.

8. You are now ready to perform the titration. This process goes faster if one person adds drops of NaOH solution while another person operates the calculator and enters volumes.

   a. Select START to begin data collection.

   b. Before you have added any drops of NaOH solution, press ENTER and type in "0" as the NaOH volume, in drops. Press ENTER to save the first data pair for this experiment.

   c. Add one drop of NaOH solution. Be sure to hold the dropper bottle vertically to ensure that the drop size is uniform. **CAUTION:** *Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.* Stir with a toothpick to uniformly mix the solution. When the pH stabilizes, press ENTER and enter "1" as the number of drops of NaOH solution added. You have now saved the second data pair for the experiment.

   d. Add a second drop of NaOH solution and stir. When the pH stabilizes, press ENTER, and enter "2" as the number of drops added.

   e. Continue this procedure until 20 drops of NaOH solution have been added.

9. Press STO▸ when you have finished collecting data.

10. Examine the data on the displayed graph to find the *equivalence point*—that is, the 1-drop volume increment that resulted in the largest increase in pH. As you move the cursor right or left on the displayed graph, the volume (X) and pH (Y) values of each data point are displayed below the graph. Go to the region of the graph with the large increase in pH. Find the NaOH volume (in drops) just *before* this jump. Record this value in the data table. Then record the NaOH volume *after* the drop producing the largest pH increase was added.

11. (optional) Print a copy of the graph of pH *vs*. volume.

12. Carefully loosen the pH Sensor from the utility clamp and dispose of the beaker contents as directed by your instructor. Thoroughly rinse out the microbeaker and the pH Sensor tip. Remove the microbeaker from the sensor. Rinse the sensor tip with distilled water and return it to the pH Sensor storage bottle.

PROCESSING THE DATA

1. Use your printed graph to confirm the volume of NaOH titrant you recorded *before* and *after* the largest increase in pH values upon the addition of 1 drop of NaOH solution.

2. Determine the volume of NaOH added at the equivalence point. To do this, add the two NaOH values determined above and divide by two (use 0.5-drop increments in your answer).

3. Using the formula in the introduction of the experiment, calculate the concentration of the hydrochloric acid solution (in M or mol/L).

DATA AND CALULATIONS TABLE

Concentration of NaOH	M
NaOH volume added *before* the largest pH increase	drops
NaOH volume added *after* the largest pH increase	drops

Volume of NaOH added at equivalence point	drops
Concentration of HCl	M

Experiment

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TEACHER INFORMATION

Microscale Acid-Base Titration

1. This experiment can be done prior to Experiment 24, "Acid-Base Titration". Students will quickly discover the shape of an acid-base titration curve for the reaction between a strong acid and strong base. They should not expect to determine precise concentration values using this method. It is meant to be an introduction to a traditional acid-base titration, not a substitute for more precise methods.

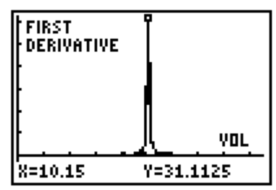
2. The preparation of 0.10 M NaOH requires 4.0 g of NaOH per liter of solution. Since the equivalence point concentrations are only approximate, using a value of ~0.10 M works well for this experiment. **HAZARD ALERT:** Corrosive solid; skin burns are possible; much heat evolves when added to water; very dangerous to eyes; wear face and eye protection when using this substance. Wear gloves. Hazard Code: B—Hazardous.

3. Unknown samples with HCl concentrations in the 0.080 to 0.100 M range work well. The preparation of 0.080 M HCl requires 6.7 mL of concentrated HCl per liter of solution. HCl that is 0.100 M requires 8.4 mL of concentrated reagent per liter. **HAZARD ALERT:** Highly toxic by ingestion or inhalation; severely corrosive to skin and eyes. Hazard Code: A—Extremely hazardous.

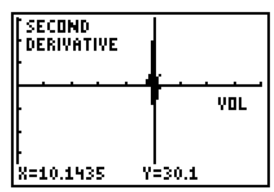
   The hazard information reference is: Flinn Scientific, Inc., *Chemical & Biological Catalog/Reference Manual, 2000*, P.O. Box 219, Batavia, IL 60510. See *Appendix F* of this book, *Chemistry with Calculators*, for more information.

4. The HCl and NaOH solutions can be dispensed from microscale Beral pipets if you do not have dropper bottles.

5. An alternate way of determining the precise equivalence point of the titration is to take the second derivative of the pH-volume data. When DataMate is transferred to the calculator, a small program called PHDERIVS will be copied into calculator as well.[1] PHDERIVS allows you to view first and second derivative plots of pH-volume data. PHDERIVS is set up to analyze volume data in L1 and pH data in L2. To run the program, follow this procedure:



   a. After you have collected pH-volume data, leave DataMate by selecting QUIT from the main screen.
   b. Start the PHDERIVS program. **Note:** On a TI-83 Plus, PHDERIVS will be a loaded as a program (press PRGM, not APPS). On a TI-86, 89, 92, or 92 Plus, the PHDERIVS program will be listed alphabetically below the DATAMATE programs.
   c. Proceed to the GRAPHS menu. Select SECOND DERIV, a plot of Δ^2pH/Δvol^2.
   d. Using the arrow keys, move the cursor to the equivalence point. This will be the point where the curve crosses the zero line. The x-value shown at the bottom of the screen is the volume of acid at the equivalence point.



[1] PHDERIVS will not be loaded onto the TI-82, TI-83, and TI-73 calculators because of their memory capabilities. If you would like a copy of PHDERIVS for these calculators, visit our web site: www.vernier.com/cbl/progs.html

6. The stored Vernier pH calibration will work fine for this experiment.

7. You can purchase additional pH storage solution bottles from Vernier Software to use as the microbeakers. Simply cut the bottles in half to use in this experiment. Distribute the half with the threaded opening to student lab stations. You do not need to include the cap, since students are instructed to use the cap and O-ring already on the probe). Order information is:

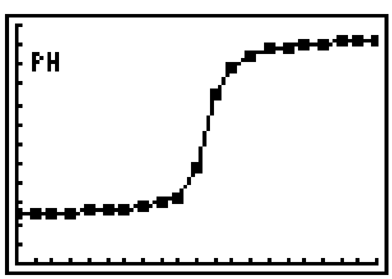
   pH Storage Solution Bottles pkg of 5 order code: BTL $10.00

8. Explain to your students the purpose of adding the phenolphthalein indicator. They can easily observe the color change and large pH increase occur simultaneously in this experiment.

SAMPLE RESULTS

Concentration of NaOH	0.10 M
NaOH volume added before largest pH increase	10 drops
NaOH volume added after largest pH increase	11 drops

Volume of NaOH added at equivalence point	$\frac{10 + 11}{2}$ = 10.5 drops
Concentration of HCl	= 0.10 M x $\frac{10.5 \text{ drops}}{10 \text{ drops}}$ = ~0.105 M



Microscale titration for sodium hydroxide and hydrochloric acid

Appendix

A

Using the CD

Important: You do not need to use the CD included with this book unless you want to change something in the student instructions to meet your needs.

The CD located inside the back cover of this book contains two folders:

- **Chemistry w Calculators Word** - For use with this book. Supports the DataMate calculator program with LabPro or CBL 2. Contains files for each of the 36 student experiments in this book.
- **Chemistry w Computers Word** - Not for use with this book. Supports Logger *Pro* computer software with LabPro, Serial Box Interface, or ULI. Contains files for 31 student experiments in the lab book, *Chemistry with Computers*.

Using the *Chemistry with Calculators* Word-Processing Files

Start Microsoft Word, then open the file of your choice from the Chemistry with Calculators Word folder. Files can be opened directly from the CD or copied onto your hard drive first. These files can be used with any version of Microsoft Word that is Version 6 or newer.

All file names begin with the experiment number, followed by an abbreviation of the title; e.g., 01 Endo- Exothermic is the file name used for *Experiment 1, Endothermic and Exothermic Reactions*. This provides a way for you to edit the tests to match your lab situation, your equipment, or your style of teaching. The files contain all figures, text, and tables in the same format as printed in *Chemistry with Calculators*.